

Non fatty foods — Determination of N-methylcarbamate residues —

Part 2: HPLC method with clean-up on a diatomaceous earth column

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British Standard

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National foreword

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terre de diatomées

Fettarme Lebensmittel - Bestimmung von N-
Methylcarbamatrückständen - Teil2: HPLC-Verfahren mit
Reinigung auf einer Kieselgur-Säule

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Foreword

This document (EN 14185-2:2006) has been prepared by Technical Committee CEN/TC 275 "Food analysis - Horizontal methods", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2006, and conflicting national standards shall be withdrawn at the latest by November 2006.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This draft European Standard specifies a high performance liquid chromatographic (HPLC) method for the determination of residues of N-methylcarbamate pesticides in fruits and vegetables and is based on the method of Krause [1].

The method has been validated by collaborative study for aldicarb, carbofuran, furathiocarb, methomyl, oxamyl, propoxur and thiodicarb parent compounds and for the metabolites aldicarb sulfoxide, aldicarb sulfone (aldoxycarb) and 3-hydroxy-carbofuran in tomatoes and oranges at levels between 0,04 mg/kg and 0,25 mg/kg.

No collaborative data are available for the performance of the method in the determination of other significant compounds although it is known that the method is unsatisfactory for benfuracarb.

2 Principle

The sample is homogenized with dichloromethane, the homogenate is filtered and the filtrate evaporated. For citrus fruits only, the sample is homogenized with acetonitrile, the filtrate is washed with n-hexane and evaporated. In both cases, the residue is dissolved in an aqueous sodium chloride solution. The solution is transferred to a diatomaceous earth column and the column is eluted with dichloromethane. The eluates are evaporated and the residue is dissolved in methanol. In this solution, the N-methylcarbamates are determined by reversed-phase high-performance liquid chromatography (HPLC) with post-column hydrolysis. The methylamine formed is allowed to react with o-phthalaldehyde and 2-mercaptoethanol and the derivatives are detected with a fluorescence detector [2].

3 Reagents

3.1 General

Unless otherwise specified, use reagents of recognized analytical grade, preferably for HPLC and pesticide residue analysis, and distilled water or water of equivalent purity.

Label all standard containers with name and purity of the pesticides. For the full chemical names and structures, see ISO 1750.

3.2 Safety aspects

WARNING — The use of this standard may involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

3.3 Acetonitrile

3.4 n-Hexane

3.5 Acetonitrile, saturated with n-hexane

In a separating funnel, shake 400 ml of acetonitrile (3.3) with 40 ml of n-hexane (3.4) for 3 min and use the lower layer.